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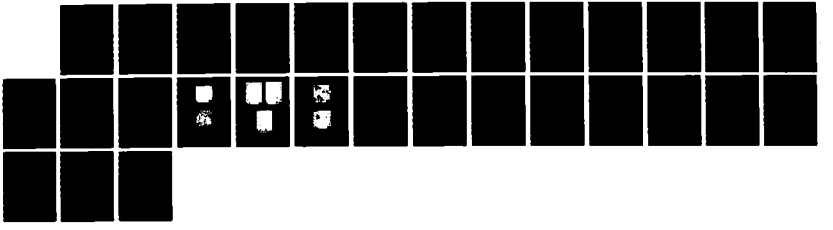

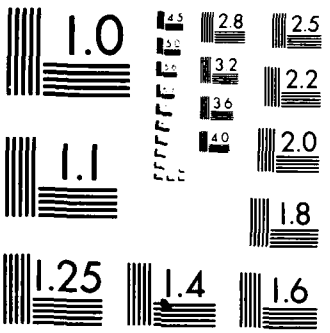

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The ambient and elevated temperature strength and microstructural stability of powder processed Al-Fe-Ni alloys are being evaluated with respect to processing mode, microstructure, and microstructural stability. The overall objective is to establish a basic understanding of processing-microstructure relations in this new class of alloys in order to establish design guidelines for limiting stresses and temperatures.

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The data and observations confirm a significant enhancement in strength and microstructural stability compared to the same alloy without mechanical alloying. Currently, creep studies are in progress on the mechanically alloyed material, and microstructural stability is being assessed above 500°C.

ANNUAL TECHNICAL REPORT

"A FUNDAMENTAL STUDY OF P/M PROCESSED ELEVATED TEMPERATURE ALUMINUM ALLOYS"

AFOSR Grant #82-0010

Principal Investigators: A. Lawley and M.J. Koczak

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October 1985

ABSTRACT OF RESULTS

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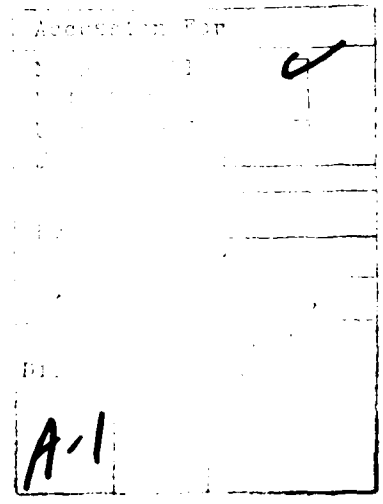

INTRODUCTION

In aerospace structural applications, there is a need for high performance aluminum alloys with improved properties and elevated temperature stability. A goal of the Air Force is to develop aluminum alloys for long-time service in the temperature range 230-340°C. There are severe inherent limitations to these property and performance goals utilizing ingot metallurgy (I/M) and this has stimulated research into powder metallurgy (P/M) as a processing alternative (1,2). Of particular promise and potential are the P/M technologies of rapid solidification (RS) and mechanical alloying (MA) (3-5). It has been clearly demonstrated that P/M processing provides enhanced alloying flexibility and results in fine-scale homogeneous microstructures with minimal attendant solute segregation (6,7).

The key to improved high temperature strength is the presence of a fine-scale thermodynamically stable dispersion, uniformly distributed in the alloy matrix. Transition metal elements are added to aluminum to produce dispersions of stable, hard intermetallics (8,9) which are resistant to coarsening. Specifically, Co or Fe and Ni give rise to dispersiods of Co_2Al_9 or $FeNiAl_9$ (10-12). The degree of homogeneity, size, and volume fraction of the dispersoid are limited in I/M alloys by virtue of the inherent low rates of solidification. P/M processing involves rapid solidification of the air atomized melt which ensures a uniform, fine-scale dispersoid in the aluminum matrix. This results in a significant increase in strength by virtue of (i) extending solid solubility and thereby increasing the volume fraction of dispersoid, and (ii) a fine stabilized grain structure of grain size ~1μm.

THE PRESENT PROGRAM

In the first phase of this program, we have established optimum conditions for the P/M processing, resulting microstructures and mechanical properties of prealloyed Al-Fe-Ni (13,14). Three volume fractions of $FeNiAl_9$ dispersoid were studied, namely 0.19, 0.25 and 0.32. Ambient and elevated temperature tensile and creep response were assessed at temperatures up to 400°C and microstructural stability was evaluated at

temperatures up to 500°C.

In order to further enhance the properties and performance of this new generation of P/M aluminum alloys, rapidly solidified powders have been subjected to mechanical alloying (MA) prior to hot consolidation (5,15,16). MA was developed by Benjamin (3) in order to prepare oxide dispersion strengthened superalloys. The process employs a high-energy ball mill. Stearic acid and methanol are added to the powder charge as a process-control agent (17) to prevent excessive welding of the powder to itself, and to the steel balls. The ball milling operation results in the development of a very fine, uniform submicron dispersion of oxides and carbides in the aluminum matrix coupled with a fine grain size of about 0.5μm.

This progress report summarizes studies to-date in which the prealloyed powder was mechanically alloyed prior to consolidation. Ambient and elevated temperature tensile response and microstructural stability have been examined up to 500°C.

PROGRAM SUMMARY

(a) Procedures

Al-Fe-Ni powder of nominal weight composition Al-4.9% Fe-4.8% Ni was air atomized by Alcoa. This composition falls within the two-phase Al + $FeNiAl_9$ region of the ternary phase diagram, according to the room temperature isotherm. The powder contains 0.25 volume fraction of $FeNiAl_9$ as the intermetallic dispersoid.

The prealloyed atomized powder was blended with pure aluminum powder and mechanically alloyed at Novamet to give a final volume fraction 0.19 of $FeNiAl_9$. The necessary organic process control agent was added by Novamet.

MA powder was cold isostatically pressed in aluminum cans, outgassed, sealed and hot extruded to full density at a constant extrusion ratio of 16:1 to give a final diameter of 25.4 mm. Three combinations of outgassing temperature and extrusion temperature were used in the range 370°C to 490°C; the combinations are designated; a-processing

(low degassing temperature/high extrusion temperature); b-processing (low degassing temperature/low extrusion temperature; and c-processing (high degassing temperature/low extrusion temperature).

Material hot consolidated via each of the three processing regimes was examined by optical microscopy (OM) and transmission electron microscopy (TEM). Some of the extruded alloy was exposed at temperatures up to 500°C with time as a variable, prior to characterization by OM and TEM.

Sample preparation for OM involved grinding, diamond polishing and etching using Keller's reagent for 8 to 10 seconds. For TEM, a Phillips STEM 400 was used with an accelerating voltage of 100-120 Kv. Sample preparation consisted of slicing with a diamond wheel to a section thickness of approximately 1 mm. These sections were then surface ground to a thickness of ~0.004 mm. Disks, 3 mm in diameter, were stamped out for subsequent thinning via electrolytic jet polishing. The electrolyte consisted of 75% methanol and 25% nitric acid and was kept at a temperature of -40°C.

Room temperature hardness (Rockwell B scale) was measured on the as-extruded alloy and following elevated temperature exposure. Tensile tests were performed on a model TT-C Instron. Threaded end specimens with a 32 mm gage length and a 6.35mm gage diameter were used with the tensile load applied parallel to the direction of extrusion. Tensile tests were conducted to fracture in air at a strain rate of 2.2×10^{-4} sec^{-1}. For the high temperature tests at 200°C, 300°C, 350°C and 400°C, a dual elliptical reflecting split furnace was used; temperatures were measured with an accuracy of ±2°C.

(b) Results and Observations

(i) Microstructure

Representative microstructures of the as-extruded MA Al-Fe-Ni alloy following c-processing are shown in Figure 1. Fine oxides and carbides, uniformly distributed within the aluminum matrix between the $FeNiAl_9$ dispersoids are resolved by TEM, Figure 1(b). The average intermetallic dispersoid particle size is approximately 0.18μm which is

slightly larger than the size in the non-MA alloy (~0.11μm). Similar microstructures are observed following a-processing and b-processing. In each case, the matrix subgrain sizes are comparable, with an without MA (13,14).

Microstructurally, the major difference between the MA and non-MA Al-Fe-Ni alloys is the resistance to coarsening of the intermatllic dispersoid. At 450°C, considerable coarsening of the non MA Al-Fe-Ni is observed, accompanied by grain growth (13,14). In contrast, after long-time exposure at 450°C there is only limited coarsening of the microstrcture of the MA alloy, cf. Figures 1(a), 2(b) and 2(c). Even at 500°C, only minimal coarsening of the $FeNiAl_9$ occurs, cf. Figures 1(a) and 2(a). Corresponding TEM comparisons of the microstructure are illustrated in Figure 3 for the a-processed alloy. These confirm the excellent microstructural stability at 450°C induced by MA (13,14).

Currently, the MA material is being subjected to long-time exposure at temperatures of 550°C and 610°C. From a limited TEM study, it appears that MA promotes a significant improvement in resistance to coarsening of the $FeNiAl_9$ intermetallic, compared to the non-MA condition.

(ii) Hardness and Tensile Properties

Room temperature hardness after isochronal elevated temperature exposure is shown is Figure 4 for the three processing conditions examined. For comparison, the response of the non MA alloy (containing the same volume fraction (0.19) $FeNiAl_9$) is included. It is seen that after exposure above 400°C, the room temperature hardness of the non-MA material is lowered precipitously. In comparison, the MA material exhibits a temperature advantage of about 100°C; even above 500°C the room temperature hardness decreases gradually with increasing temperature of exposure. The combination of a low degassing temperature and low extrusion temperature (b-processing) promotes the highest degree of microstructural stability, Figure 4.

Hardness response, measured at room temperature following isothermal exposure

at 450°C is illustrated in Figure 5. These data confirm the superiority of the b-processing conditions. After 624 hours at 450°C, the room temperature drop in hardness of the MA b-processed alloy is only ~10%. Without MA, the corresponding decrease is about 70%, Figure 6.

Tensile repsonse also reflects the microstructural stability associated with MA. The temperature dependence of yield and tensile strength is compared in Figure 7 for a, b and c-processing. Data for the non MA alloy are included for comparison. Consistent with the hardness data, the tensile data show the superiority of b-processing, at least up to about 350°C. The effect of processing mode is more pronounced at the lower temperatures. Above about 350°C, processing history has only a small effect on yield and tensile strength. The strength of the non MA alloy is lower than that of the MA alloy at all test temperatures, Figure 7.

The increase in strength brought about by MA is accompanied by a decrease in ductility, as measured by strain to failure in the tensile test, Figure 8. Quantitatively, MA increases ambient temperature yield strength by about 77% (Figure 7) but there is a decrease in ductility of about 85% (Figure 8). The extent of work hardening is reduced dramatically by MA, particularly in the lower temperature range.

The effect of isothermal exposure at 450°C on strength at room temperature and 250°C is illustrated in Figure 9 for the c-processed alloy. A comparison with Figure 7 shows that strength is only slightly impaired after long time exposure (>600 hours) at 450°C. The effect of processing mode on room temperature strength following isothermal exposure at 450°C is shown in Figure 10; this figure is the analog of the hardness response shown in Figure 5. Strength retention is clearly superior following b-processing.

Creep tests have recently been initiated on the MA Al-Fe-Ni alloy. It is planned to cover the temperature range from 250 to 400°C at stress levels in the range of about 75 MPa to 150 MPa. A comparison with the creep response of the non-MA alloy is also inlcuded in the on-going study.

(c) Interpretation and Significance of Results

Coarsening of the $FeNiAl_9$ intermetallic requires the diffusion of Fe and Ni atoms, either via the subgrain boundaries or through the matrix. Since the activation energy for grain boundary diffusion is lower than that for volume diffusion, the former should predominate at the lower temperatures. The present TEM observations (Figures 1(b), 3(a) and 3(b)) confirm that MA introduces a fine-scale dispersion of oxides/carbides. These are present in the matrix, at matrix-intermetallic interfaces, and at matrix subgrain boundaries. The oxides/carbides on the subgrain boundaries are expected to inhibit diffusion along such paths by acting as vacancy sinks.

The oxides/carbides at matrix-intermetallic $FeNiAl_9$ interfaces in the MA alloy are considered to hold the key to enhanced microstructural stability. While the mechanism is not clear, the fine-scale oxides and/or carbides at the matrix-intermetallic interfaces must interfere with the diffusion of Fe and Ni atoms. In consequence, coarsening of the intermetallic is inhibited. It has been shown (13) that in the absence of the fine-scale oxides/carbides in the non-MA material, Ostwald ripening of the intermetallic proceeds rapidly at 450°C. The contribution to coarsening from matrix diffusion of Fe and Ni will increase with increasing temperature.

Singer et 1l (17) have confirmed the presence of Al_2O_3 and Al_4C_3 in MA aluminum. The size and morphology of the oxide/carbide dispersions observed in the present study is similar to that reported by Singer et al (17). Other oxides and/or carbides may form at the higher exposure temperatures. It is difficult to distinguish between the oxides and the carbides in the MA material.

In dispersion-strengthened alloy systems, recrystallization is strongly inhibited by the pinning of subgrain boundaries by dispersoids (18-20). In the hot-extruded MA material, both the $FeNiAl_9$ intermetallic dispersion (size ~0.18μm) and the fine-scale oxide/carbide dispersion (size ~30nm) act to pin subgrain boundaries. At 450°C the

oxides/carbides severely restrict coarsening of the intermetallic and the subgrain boundaries remain pinned. Above 500°C general coarsening occurs, with an attendant release of the subgrain boundaries. In the non MA alloy coarsening of the $FeNiAl_9$ intermetallic occurs at 450°C and the subgrain boundaries are no longer as effectively pinned.

The changes in room temperature hardness following elevated temperature exposure of the MA alloy are understood in terms of the accompanying microstructural modifications. Thus, a combination of dispersoid coarsening, recrystallization and subsequent grain growth at temperatures above about 450° lead to a decrease in hardness.

In summary, the fine dispersion plays a dual role in the hardening mechanism, first by promoting dispersion hardening and secondly by inhibiting the coarsening of the intermetallic dispersion. The ambient temperature strength of the alloy can be described by the superposition of dispersion strengthening and subgrain boundary strengthening (21). In the Al-Fe-Ni system, as temperature increases, a decrease in the aluminum matrix flow stress occurs by subgrain boundary and matrix weakening. The higher strength exhibited by the MA alloy is a result of dispersion hardening, inhibition of coarsening , and stabilization and strengthening of the subgrain boundaries by the fine oxide/carbide dispersion.

Above a threshold stress dislocations can evade pinning dispersoid particles by particle shearing, bowing via the Orowan mechanism, or by climb. Since the $FeNiAl_9$ and oxide/carbide particles are hard/non-deformable, and there is no evidence of dispersoid/particle shearing (13), deformation must proceed by the dislocation interparticle bowing mechanism. This process can be assisted by thermal activation at high temperatures. Strength increases with decreasing interparticle distance, consistent with the introduction of the fine scale, uniform distribution of oxides and carbides during MA, Figures 1(b) and 3. Microvoid nucleation at particle-matrix interfaces is enhanced by

a decrease in interparticle spacing. These small voids coalesce and this reflects in a lower strain to fracture. This mechanism accounts for the decrease in ductility resulting from MA, Figure 8.

With increasing temperature, dislocations can climb and bow more easily through the weakened matrix, assisted by diffusion. The transition in mechanism from Orowan looping to dislocation climb occurs gradually with increasing temperature. The oxide particles and carbides in the MA alloy are extremely fine (~30nm) and these present only a small barrier to dislocation climb. Thus, the fine oxides and carbides introduced by MA become ineffective barriers to climb above about 300°C and the strength in the MA and non MA alloys is therefore similar.

In terms of microstructure (TEM), no significant differences are detected as a function of processing regime, i.e. a, b, or c-processing. Clearly, however, differences in strength and microstructural stability exist in the a, b and c-processed MA alloy.

Overall superiority achieved via b-processing (i.e. low degassing temperature) implies that adequate degassing is achieved. Insufficient degassing results in microporosity since voids form as a result of the liberation of hydrogen/hydrocarbons from adosrbed moisture on powder particle surfaces, and from the MA process control agent. The low extrusion temperatures used in the b-processing mode minimize in-situ coarsening of the intermetallic, oxides and carbides. This enhances strength and increases the uniformity of the microstructure in the fully-densified MA alloy.

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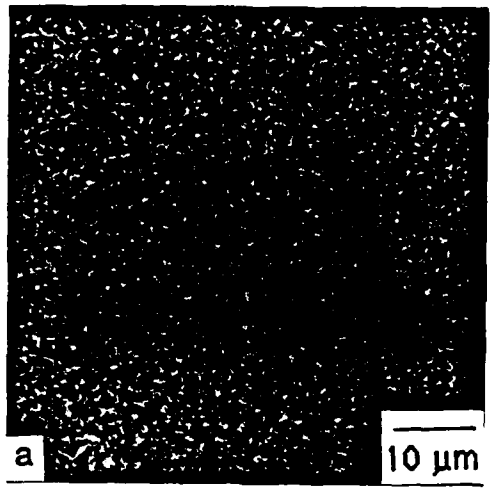


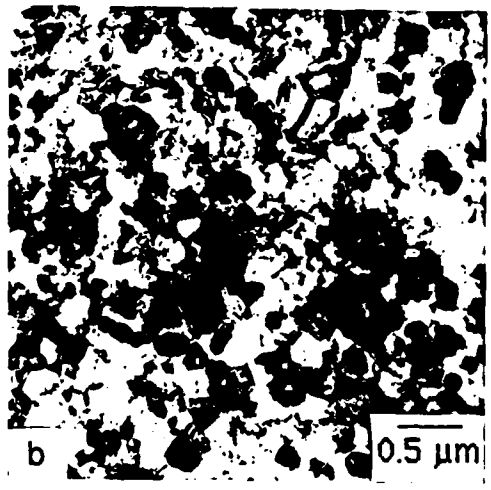


Figure 1: Optical and TEM micrographs. c-processing, as-extruded condition.

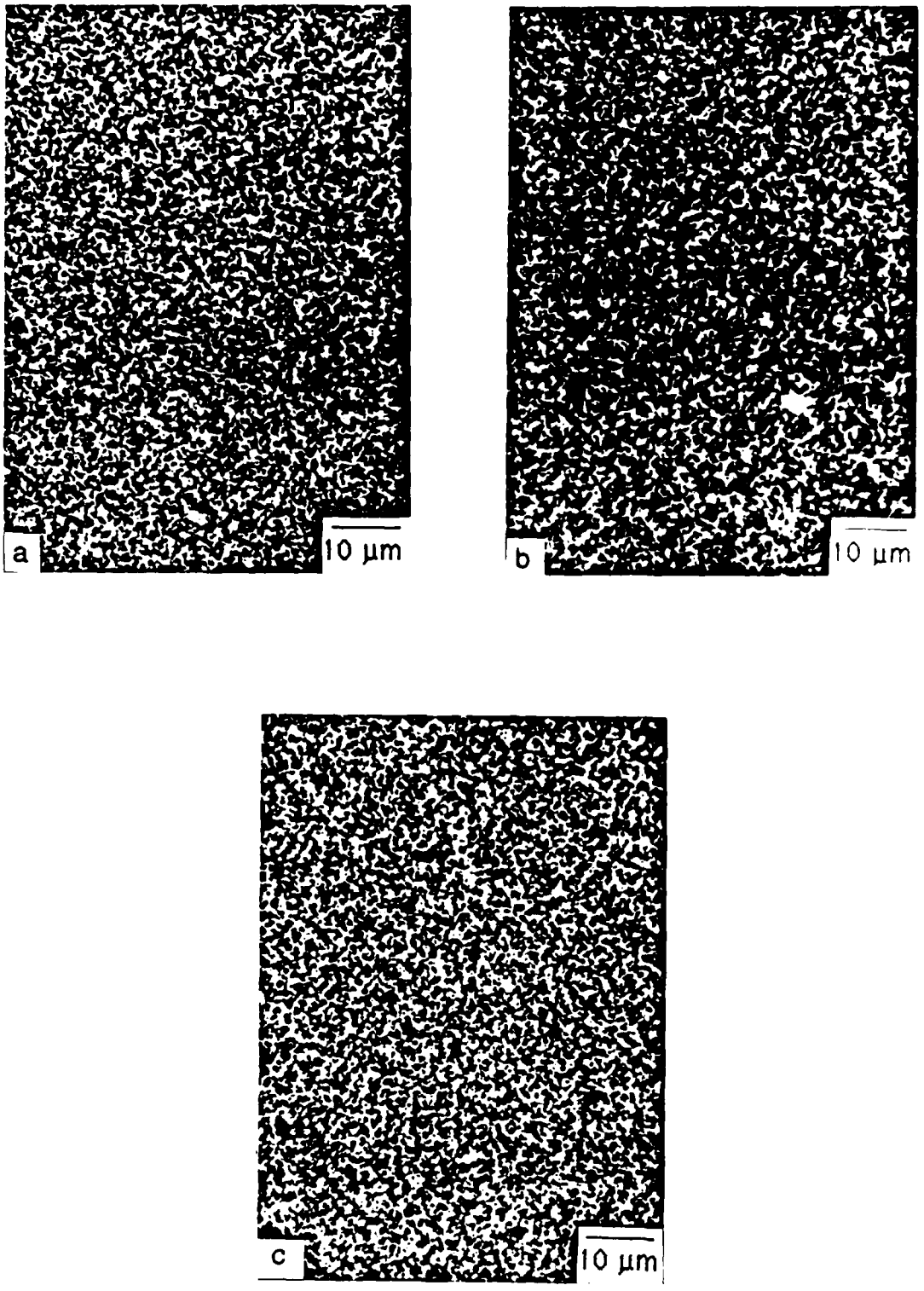
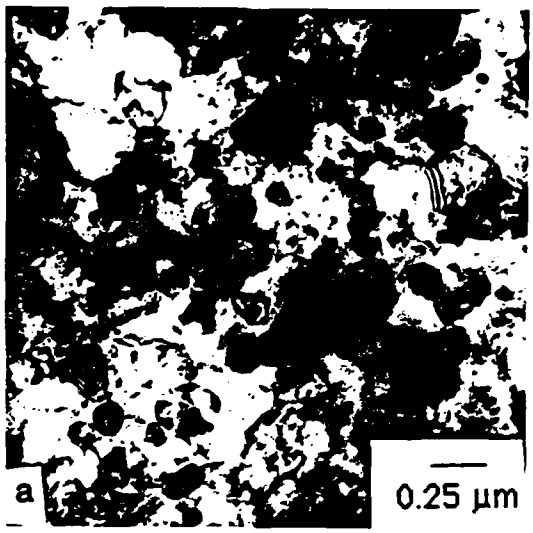


Figure 2: Optical micrographs following elevated temperature exposure (a) c-processing, 500°C/hr. (b) b-processing, 450°C/288 hrs. (c) c-processing, 450°C/288 hrs.



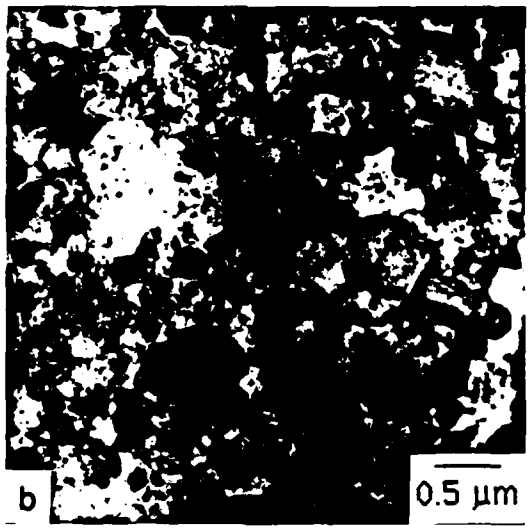


Figure 3: TEM. a-processing. (a) As-extruded, (b) 450°C/288 hrs.

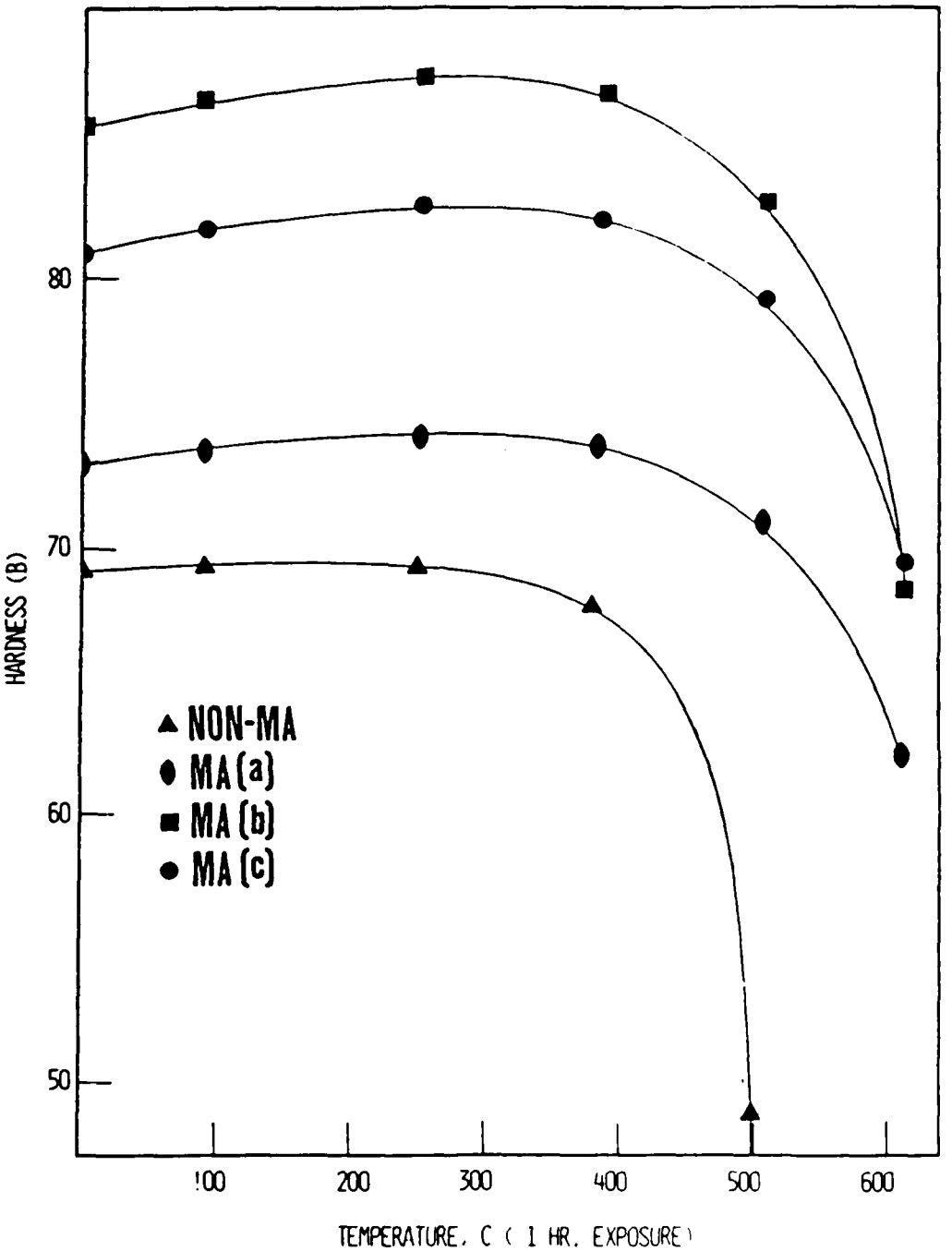


Figure 4: Room temperature hardness after elevated temperature (isochronal) exposure.

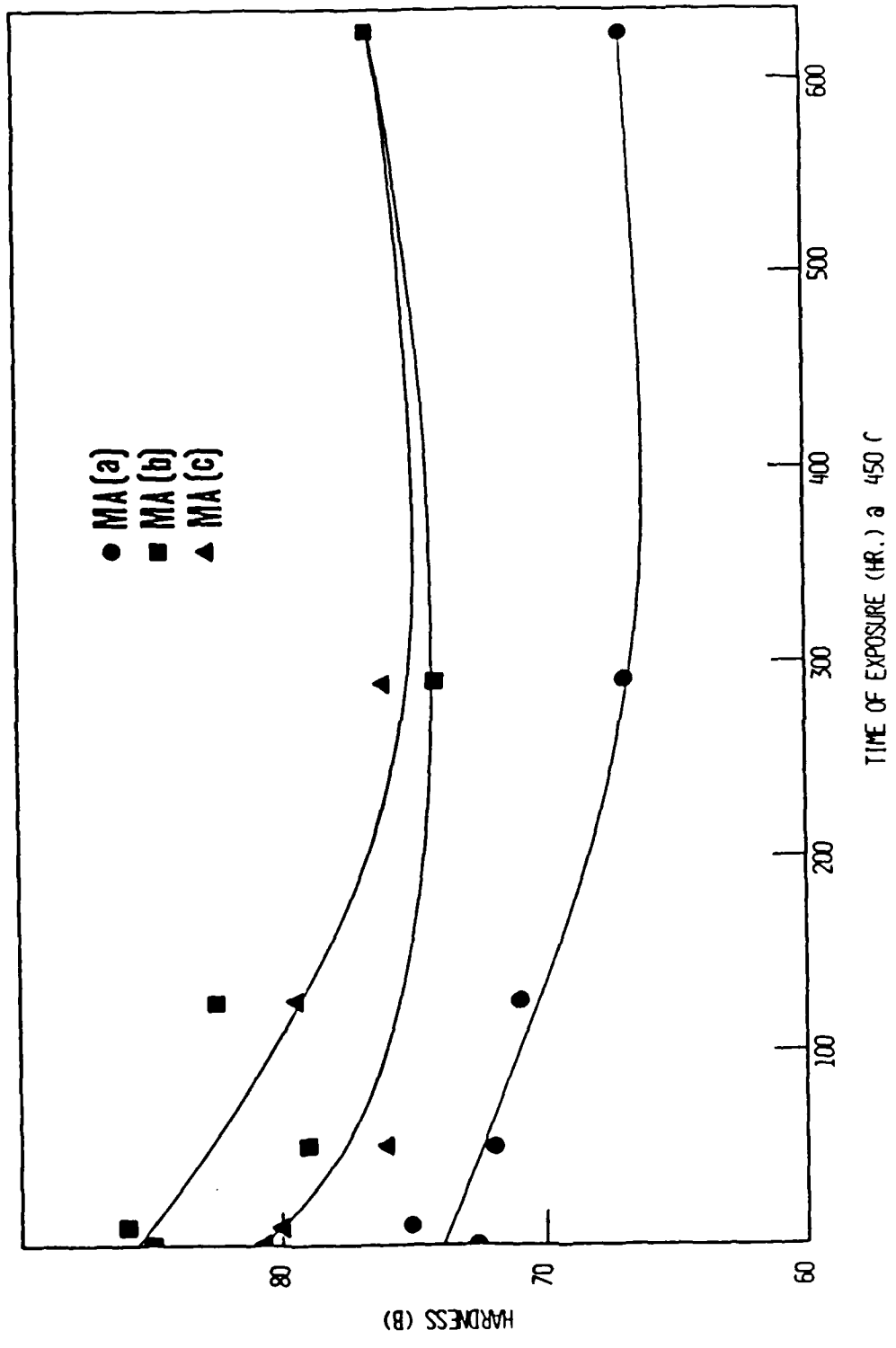


Figure 5: Effect of processing mode (a, b or c) on room temperature hardness, following isothermal exposure.

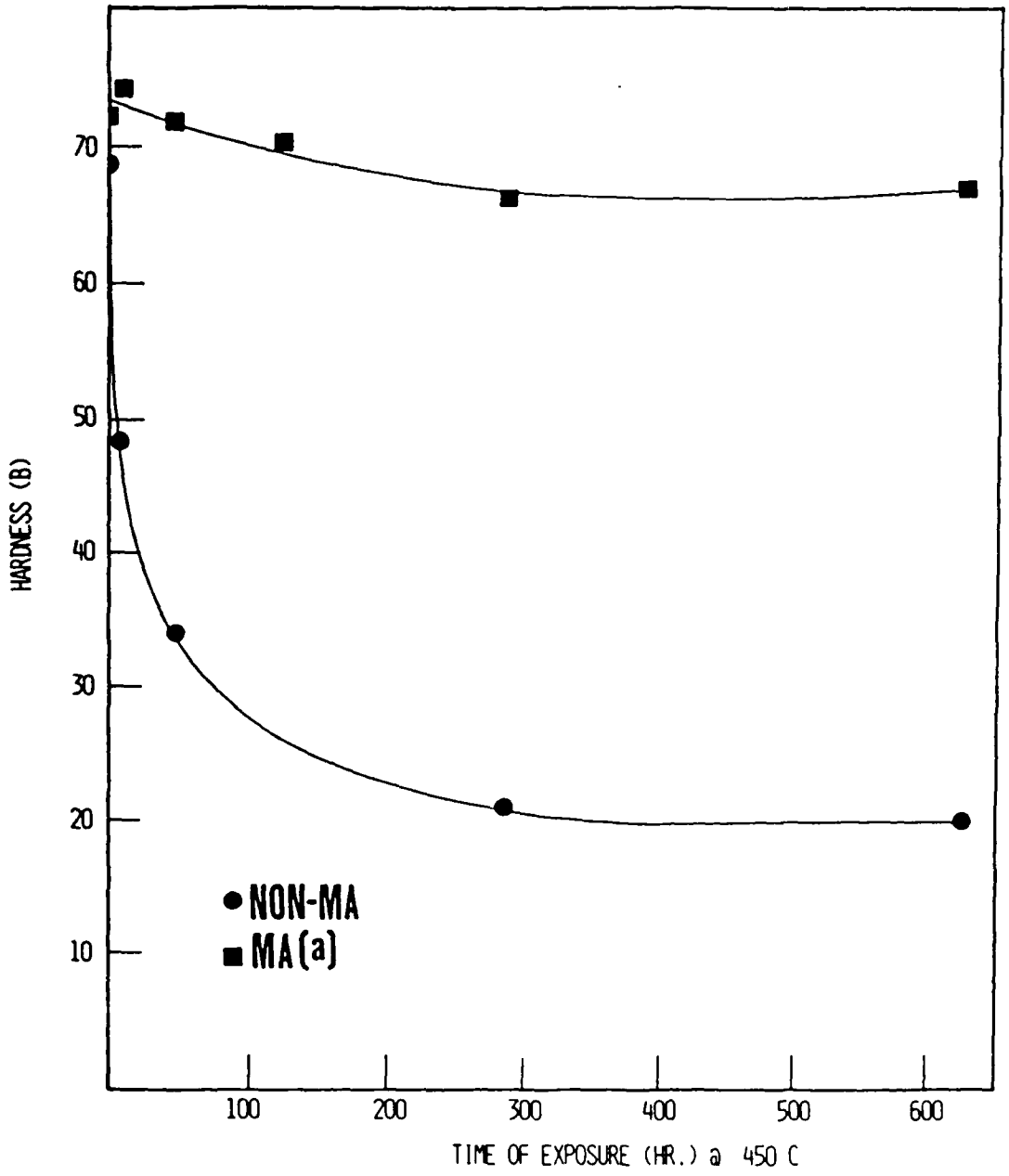


Figure 6: Effect of mechanical alloying on room temperature hardness, following isothermal exposure.

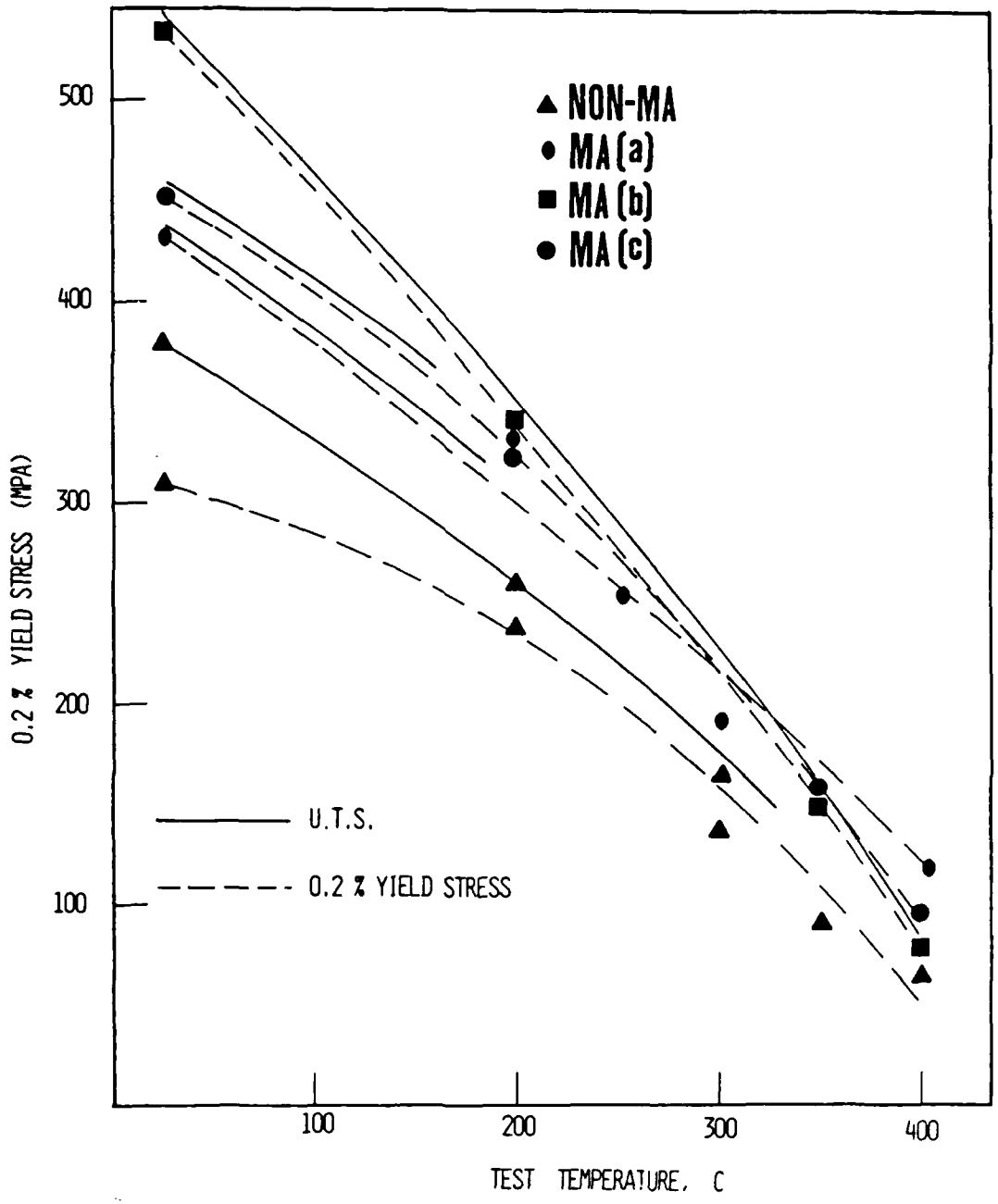


Figure 7: Temperature dependence of yield and tensile strength.

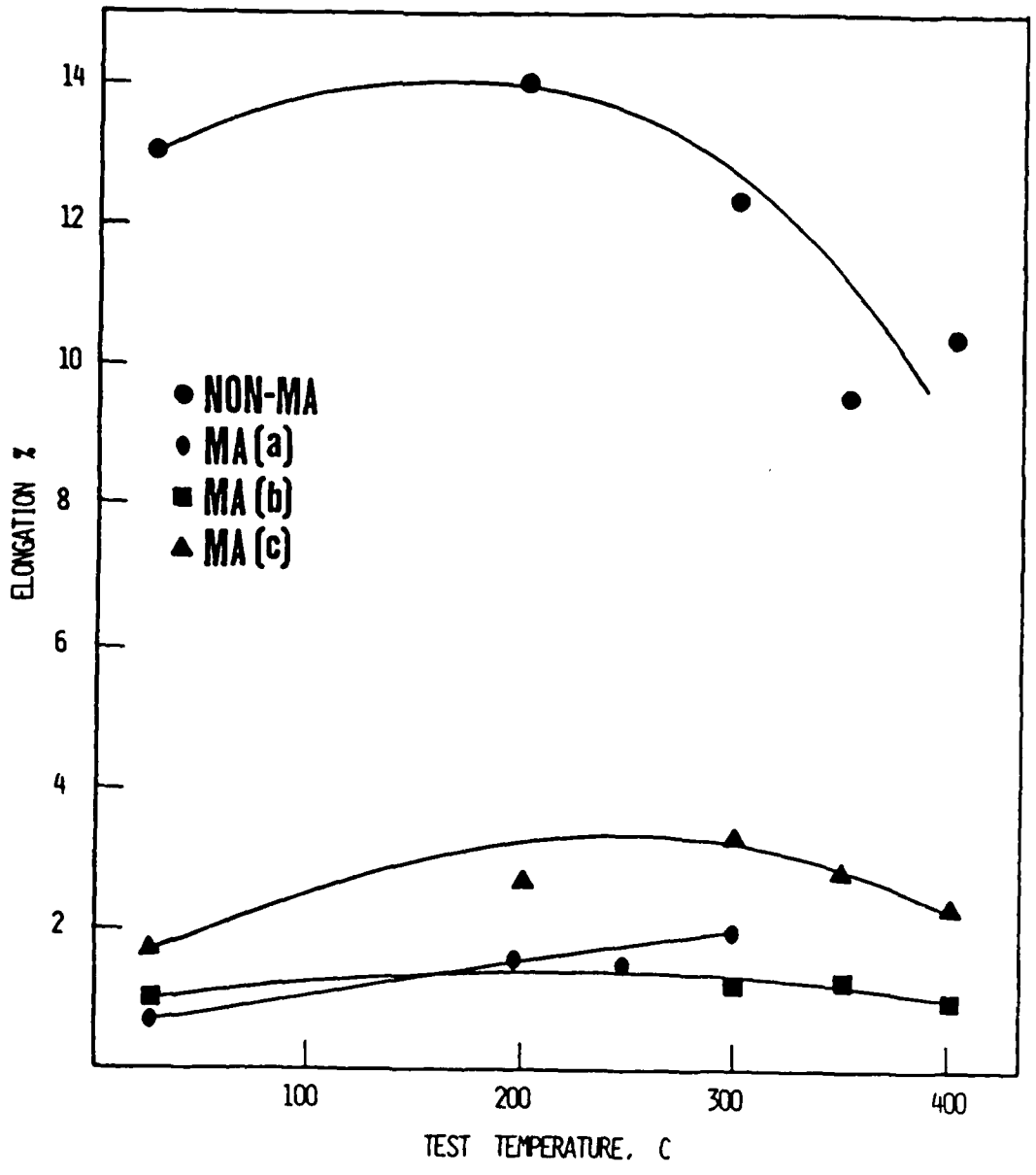


Figure 8: Temperature dependence of ductility

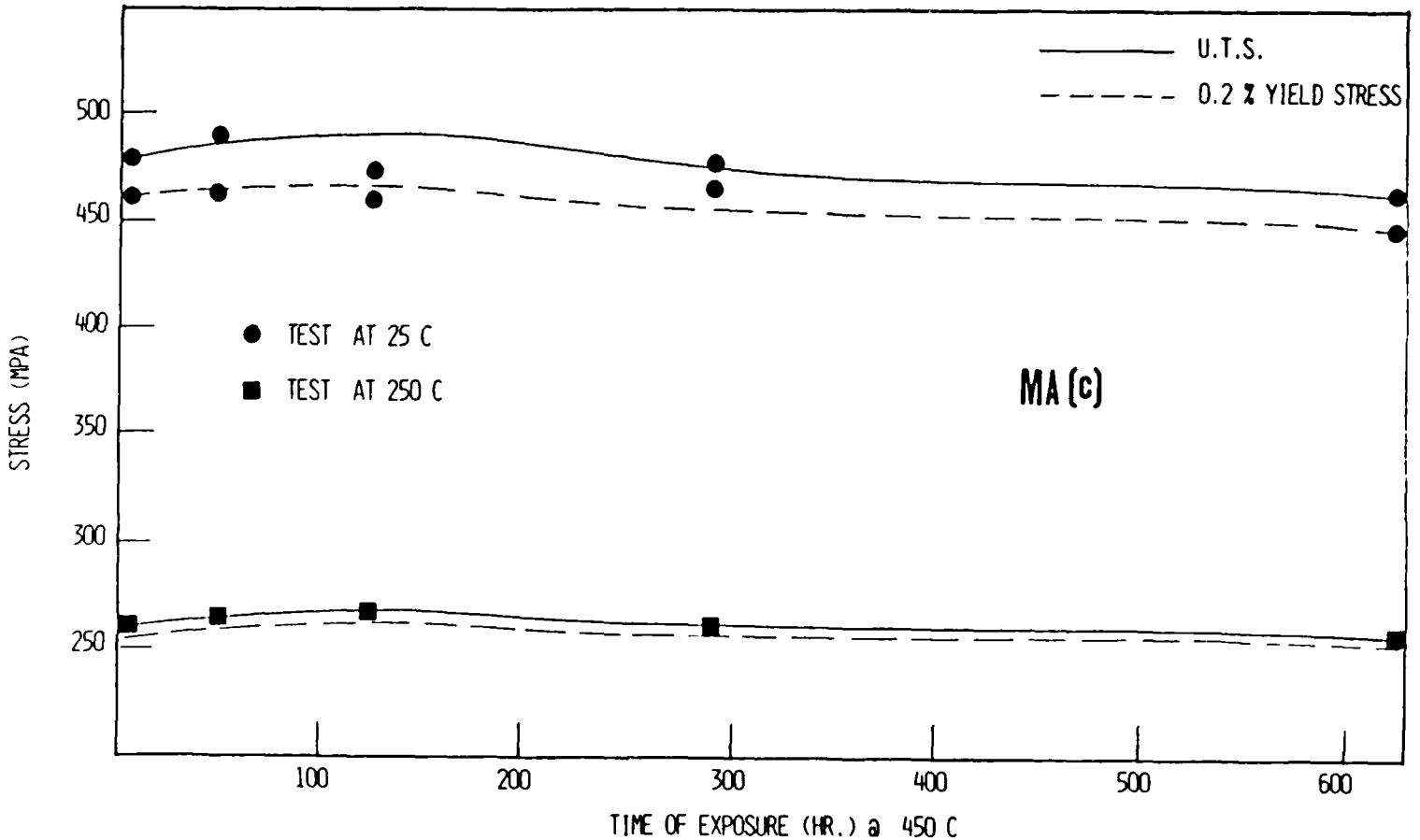


Figure 9: Effect of exposure time at 450°C on strength at room temperature and 250°C. c-processing.

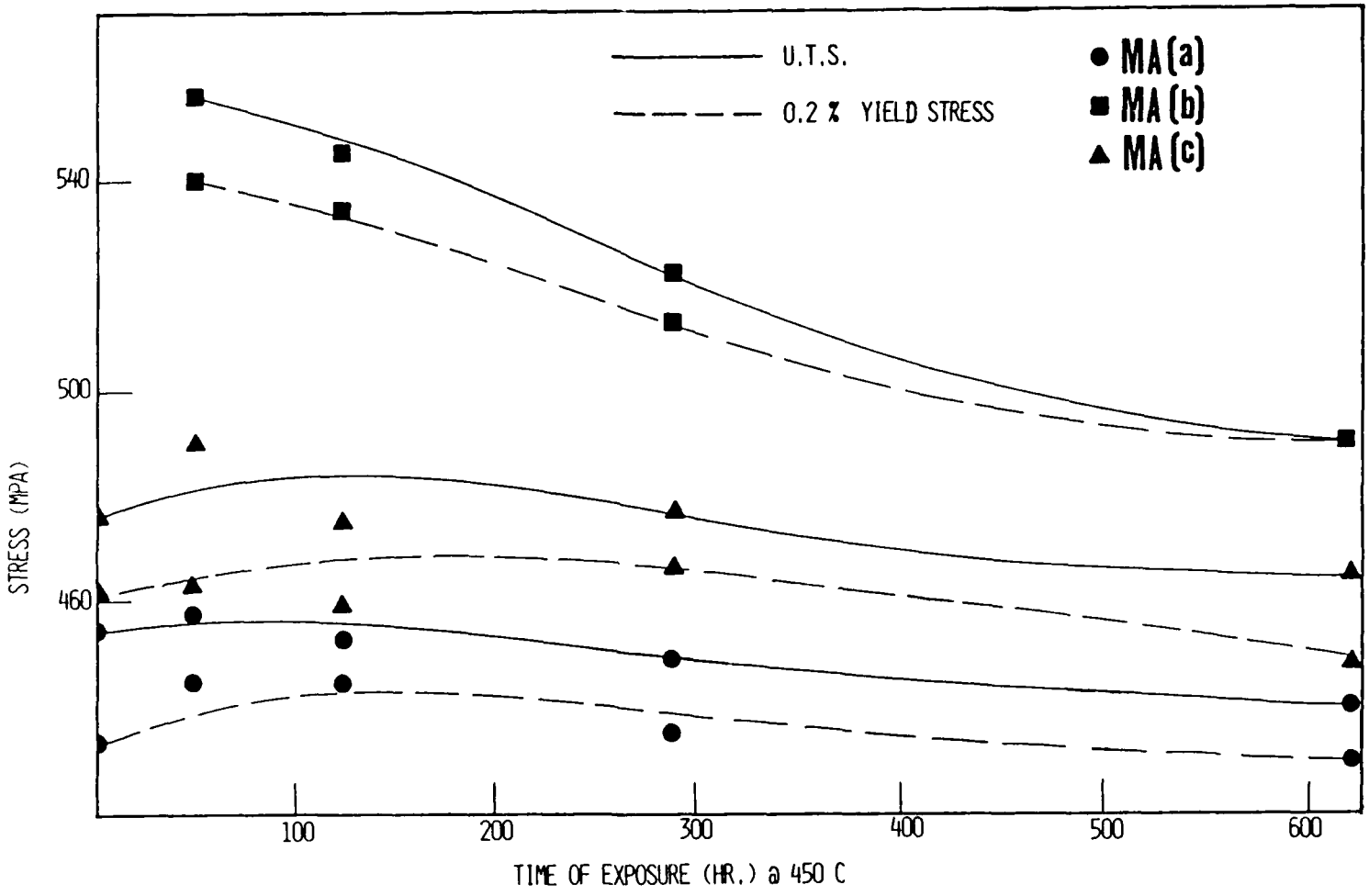


Figure 10: Effect of processing mode (a, b or c) on room temperature strength, following isothermal exposure.

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High Strength Powder Metallurgy Aluminum Alloys II, Editors: G.J. Hildeman and M.J. Koczak, The Metallurgical Society, Inc., in press.

PERSONNEL

A. Lawley - Professor and Co-Principal Investigator

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COUPLING ACTIVITIES

a) Presentations

"Powder Metallurgy Processing of Dispersion Strengthened Light Metal Alloys", Reynolds Metals Company, Richmond, VA., January 1985.

"Modern Powder Metallurgy Science and Technology", Virginia Section, AIME, Charlottesville, VA., January 1985.

"Rapidly Solidified Materials - Current Assessment and Future Directions", AIME Annual Meeting, New York City, N.Y., February 1985.

"Rapid Solidification Science and Technology", Ford Motor Company, Dearborn, MI, April 1985.

"Aluminum Powder Metallurgy", General Electric R&D Center, Schenectady, NY, May 1985.

"Rapidly Solidified Powder Processes - Models and Mechanisms for Atomization and Consolidation", Northeastern Regional Meeting, The Metallurgical Society of AIME, Morristown, NJ, May 1985.

"The Particle Metallurgy - Rapid Solidification Interface: Atomization Models and Mechanisms", Nicholas J. Grant Symposium, Massachusetts Institute of Technology, Cambridge, MA., June 1985.

b) Technical Contacts with Other Laboratories

Both principal investigators have interacted with other research personnel engaged in similar and related research in industry, government and academia. Contacts include:

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